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EVALUATION OF FUMIGANTS FOR SHELLED CORN[1/]

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The Government grain-loan program authorized by the Agricultural Adjustment Act of 1938 led to the holding of large quantities of shelled corn in farm-type storage structures. At first conventional round galvanized-metal bins were used mostly, but later many types of wooden and metal bins, quonsets, and warehouses came into use.

When the bins were new, the only insects found in them were species that had been carried in with insufficiently cleaned corn or that had reached the bins by flight. Infestations found during the first few years consisted almost entirely of grain beetles, the immature stages of which are free living. Recently, however, there has been an increase in weevils, the grubs of which feed inside the corn kernels. In some States more than one-third of the bins contain weevil infestations. This change in the insect population with its increase of forms more resistant to fumigants, together with the new types of storage structures, has necessitated continued research to develop more effective fumigants and dosages.

Early in the program a 75-25 mixture of ethylene dichloride and carbon tetrachloride was adopted as the standard fumigant for shelled corn in steel bins for application at a dosage of 6 gallons per 1,000 bushels. Later an 80-20 mixture of carbon tetrachloride and carbon disulfide at 5 gallons per 1,000 bushels came into favor and largely replaced the one containing ethylene dichloride.

Many failures were reported with these fumigants, but usually they could be accounted for by the presence of excessive amounts of caked and damp grain on the surface or throughout the bin and also by the large amounts of dockage. Occasionally incomplete kills appeared to be associated with outbreaks of the Angoumois grain moth.

Some of the tests made with old and new fumigants under varying conditions and in various types of structures are reported herein.

1/ A portion of the tests reported herein were made in cooperation with the Grain Branch of the Production and Marketing Administration.

2/ Resigned April 13, 1951.

Technique

A number of fumigants and mixtures were tested in round metal bins and in wooden bins. Probe samples were taken before and after fumigation. In most cases 2 weeks were allowed to elapse after fumigation before samples were taken, so that the full effect of the fumigant would be realized. Eight samples were taken with a 5-foot probe--one from each quadrant, one each from the center of the top and bottom 5 feet of grain, and two horizontal surface samples from the top 3 inches, one from each half of the bin. In some tests an additional sample was taken from the center 5 feet of grain. The samples were screened to remove all free-living forms, and the grain was held for possible emergence of weevils or moths. The living and dead insects were averaged on the basis of a 1,000-gram sample.

Tests with 80-20 Mixture

The 80-20 mixture of carbon tetrachloride and carbon disulfide was tested under many conditions and in all types of storage structures. In general, a dosage of 5 gallons per 1,000 bushels was found to be adequate for the treatment of clean, dry, shelled corn in tightly caulked steel bins and for the spot treatment of isolated infestations in quonsets or similar large storage structures. In bins with much caked or moldy corn on the surface or large amounts of dockage and caked corn throughout the bin, this dosage was inadequate. Before fumigation it was necessary to remove all caked and moldy grain from the surface and turn and clean the remainder of the bin.

In wooden rectangular bins of clean, dry corn, a dosage of 7 gallons per 1,000 bushels was found necessary for effective results.

Tests with Chloropicrin and Chloropicrin Mixtures

Chloropicrin, alone and in admixture with other fumigants, was tested in steel bins of shelled corn. The corn was not in good condition. In most bins the surface grain was hot, damp, moldy, caked, and heavily infested with bran beetles. In preparation for fumigation the surfaces were stirred with a shovel and leveled.

Chloropicrin alone was sprinkled over the top of the corn with a watering can or bottle, or raked in just below the surface with a special applicator. Dosages of 3 and 4 pounds per 1,000 bushels were used in the first method, and of 2 and 2 1/2 pounds in the second. The results at all dosages were unsatisfactory.

Previous tests had indicated that better distribution of the vapor could be obtained by diluting the chloropicrin with a carrier fumigant. Consequently, tests were conducted with mixtures containing 2 pounds

of chloropicrin in 1 gallon of (1) carbon tetrachloride, (2) the 80-20 mixture of carbon tetrachloride and carbon disulfide, or (3) the 75-25 mixture of ethylene dichloride and carbon tetrachloride. All mixtures were sprayed over the surface of the grain.

Chloropicrin in admixture with carbon tetrachloride was tested at dosages of 1, 1 1/2, and 2 gallons per 1,000 bushels. The results were much better than with the chloropicrin alone, but even with the 2-gallon dosage a complete kill was not obtained.

Chloropicrin with the 80-20 mixture in dosages up to 1 1/2 gallons and with the 75-25 mixture in dosages up to 2 gallons per 1,000 bushels failed to give complete kills.

The results of these tests are given in table 1. In the first three treatments with chloropicrin alone and the first two with the chloropicrin-carbon tetrachloride mixture, the average number of insects per 1,000-gram sample was computed from two bins. All the other averages are on the basis of one bin only.

Table 1.--Effectiveness of chloropicrin and chloropicrin mixtures in the fumigation of shelled corn in steel bins, 1950

Fumigant	Dosage per 1,000 bushels	Temperature of corn (°F.)	Average number of insects per 1,000-gram sample	
			Alive	Dead
	Pounds			
Chloropicrin alone	2	96	37	9
	2 1/2	94	17	21
	3	96	10	20
		96	95	66
		73	114	22
	4	100	127	55
	Gallons			
Chloropicrin 2 lb. in 1 gal. of--				
Carbon tetrachloride	1	85	7	8
	1 1/2	90	3	9
		68	77	95
		80	55	99
	2	88	6	126
		92	6	60
80-20 mixture	1 1/4	78	6	130
	1 1/2	79	215	71
75-25 mixture	1 1/2	96	165	79
	1 3/4	97	68	35
	2	96	107	59

Tests with Methyl Bromide in Mixtures

In 80-20 and 75-25 Mixtures

The temporary scarcity of fumigants containing carbon tetrachloride, carbon disulfide, or ethylene dichloride made it desirable to investigate the possibility of increasing the effectiveness of the standard 80-20 and 75-25 mixtures by the addition of small quantities of a more toxic material. Consequently, 10 percent (by volume) of methyl bromide was added to each of these mixtures. In the first tests with the 75-25 mixture 2 percent of sulfur dioxide was also added as a warning gas.

In preliminary tests twelve 3,200-bushel metal bins of shelled corn were treated in early spring. The corn was in very poor condition--heavily infested and with a 6- to 8-inch layer of damp, caked, and moldy grain at the surface. Air temperatures ranged from 50° to 57° F. and grain temperatures from 50° to 95°.

In preparation for the fumigation the caked surface grain was broken up with shovels. Three replications of both mixtures were made with dosages of 2 and 3 gallons per 1,000 bushels. The fumigant was applied over the surface of the grain with a pump that delivered 2 gallons per minute when operating at a pressure of 50 pounds per square inch.

Samples taken 1 week after fumigation showed that a nearly complete kill had been obtained in all bins with both mixtures at both dosages. In three of the twelve bins a single live insect was found in caked surface grain.

In later tests, undertaken in warm weather, the fumigant evaporated so rapidly that some of the insects in the surface-caked grain survived. When the caked and moldy surface grain was removed before fumigation, this trouble was overcome (table 2).

On the basis of these tests, a 2-gallon dosage of either mixture is considered satisfactory. Supplies on hand can be used by adding 11 percent (by weight) of methyl bromide to the 80-20 mixture and 12.5 percent to the 75-25 mixture. By attaching a 1/4-inch standard pipe to the methyl bromide cylinder and inserting it through the bung to within 1 inch of the bottom of the drum, the required quantity of methyl bromide can be bubbled slowly into the fumigant. The mixing should be done in the open air. If desirable, 2 percent of chloropicrin can be added as a warning gas.

Tests with the 80-20 mixture plus 10 percent of methyl bromide in 3,200-bushel rectangular wooden bins of shelled corn showed that the 2-gallon dosage found satisfactory in steel bins did not always give a complete kill, but that a 3-gallon dosage was entirely satisfactory. During these tests grain temperatures ranged from 72° to 78° F. at an air temperature of 60°, and grain moistures ranged from 12 to 13 percent.

Table 2.--Effectiveness of various fumigants in the fumigation of shelled corn in 3,200-bushel metal bins, 1950

Fumigant	Dosage per 1,000 bushels	Temperature (°F.)		Number of insects per 1,000 grams (average of 9 samples)				Number of weevils emerged after 1 week
		Air	Grain	Before fumigation		After fumigation		
				Alive	Dead	Alive	Dead	
	Gallons							
80-20 mixture + 10% of methyl bromide	2	50	77-95	30	35	0	74	0
	3	50	81-87	158	64	0	148	0
75-25 mixture + 10% of methyl bromide	2	50	59-75	36	64	0	82	0
	3	50	59-60	268	51	0	231	0
	Pounds							
Methyl bromide-ethylene dibromide:								
1-1	2 1/2	82	85-92	45	24	2	82	1
	5	82	92-96	56	32	3	86	4
	7 1/2	82	78-88	47	32	17	52	50
2-3	2 1/2	74	100-101	87	60	31	56	15
	5	74	96-100	59	58	32	70	3
	7 1/2	74	74-88	45	55	37	47	50
3-2	2 1/2	74	77-89	90	54	15	83	0
	5	74	92-96	54	61	39	89	10
	7 1/2	74	68-91	129	56	40	84	0
1-4	2 1/2	82	64-71	64	17	41	55	30
	5	82	73-86	53	20	15	51	8
	7 1/2	82	89-100	123	55	19	101	3
Methyl bromide-chloropicrin, 80-20	3	75	66-81	45	2	88	18	2
	5	75	89-100	90	26	15	76	0
	7	75	72-93	62	1	69	37	0
	Gallons							
1,1-Dichloro-1-nitroethane-carbon tetrachloride, 17-83	1	80	104-106	56	17	4	39	0
	2	80	72-88	41	17	1	45	0
	3	80	77-93	44	27	1	65	0
Bromotrichloromethane-carbon tetrachloride, 10-90	3	82	88-93	61	37	1	67	3
	4	82	96-101	49	33	0	53	7

In 75-25 Mixture of Propylene Dichloride and Carbon Tetrachloride

A 75-25 mixture of propylene dichloride and carbon tetrachloride to which 15 percent of methyl bromide had been added also gave satisfactory results in metal bins at 2 gallons per 1,000 bushels.

Mixed with Ethylene Dibromide or Chloropicrin

Earlier work had shown methyl bromide in admixture with either ethylene dibromide or chloropicrin to be promising for the fumigation of clean, cool shelled corn in metal bins. Such mixtures are obtainable under pressure in cylinders that are self-emptying, so that they can be applied from outside the bin by merely opening a valve and allowing the fumigant to flow through a tube into the grain.

In all tests with these mixtures a coil of plastic tubing connected to the cylinder was buried 6 to 8 inches beneath the surface of the grain. The tubing extended in a complete circle about 2 feet from the walls of the bin with the free end across the middle. The end was plugged and holes were bored through the tubing at 3-foot intervals. By placing the cylinder of fumigant on a pair of scales it was possible to weigh in the required amount of fumigant. The mixtures caused frequent breakages in the tubing.

Mixtures containing methyl bromide and ethylene dibromide were made in the following proportions by volume: 1-1, 2-3, 3-2, and 1-4. All mixtures were used at the rates of 2 1/2, 5, and 7 1/2 pounds per 1,000 bushels.

The corn treated was in poor condition--heavily infested and caked in spots. Grain temperatures were generally high and ranged up to 101° F. Air temperatures ranged from 74° to 82° and grain moistures from 9.9 to 13.9 percent. Caked and moldy surface grain was removed before fumigation.

None of the mixtures gave satisfactory kills at the dosages used (table 2). It was obvious that they were not effective in the top 5 feet of corn, although in the middle and bottom portions of the bins the kill was usually good. The 1-1 mixture was the most effective. Results with all mixtures were rather inconsistent, probably owing to poor distribution. In many cases better results were obtained with 2 1/2 pounds per 1,000 bushels than with three times that dosage.

A limited number of tests were conducted with a mixture containing 80 percent of methyl bromide and 20 percent of chloropicrin. The bins were in very poor condition, and the surface grain was caked and moldy. The surface grain was not broken up, but the plastic tubing was buried in the grain below the crusted surface. Grain moistures ranged from 13.3 to 14.8 percent, grain temperatures from 66° to 100° F., and the

air temperature at the time of fumigation was 75°. Dosages of 3, 5, and 7 pounds of this mixture were applied in the same manner as the methyl bromide-ethylene dibromide mixtures. Practically no kill of adult insects was obtained at any dosage (table 2), although there was little emergence of weevils from samples of grain removed after treatment.

Ethylene Dibromide-Ethylene Dichloride-Carbon Tetrachloride Mixtures

A mixture containing 7.2 percent of ethylene dibromide, 29.2 percent of ethylene dichloride, and 63.6 percent of carbon tetrachloride (by weight), known as EB-5 grain fumigant, was tested in steel bins at dosages of 2, 3, and 5 gallons per 1,000 bushels. The fumigant was applied uniformly over the surface of the corn in a coarse spray. Probe samples taken 2 weeks after fumigation showed that an average of one adult insect per 1,000 grams survived fumigation at both the 2- and 3-gallon dosages, but that at the 5-gallon dosage mortality was complete. Weevils emerged from samples of grain fumigated at the two lower dosages, but not from samples fumigated at the 5-gallon dosage.

A mixture containing 18.3 percent of ethylene dibromide, 24.1 percent of ethylene dichloride, and 57.6 percent of carbon tetrachloride was found to be no more efficient.

1,1-Dichloro-1-nitroethane-Carbon Tetrachloride Mixture

A mixture containing 2 pounds of 1,1-dichloro-1-nitroethane in 1 gallon of carbon tetrachloride (approximately 17-83 percent by volume) was tested at dosages of 1, 2, and 3 gallons per 1,000 bushels. The fumigant was applied in a coarse spray uniformly over the surface of eight 3,200-bushel bins. The caked and moldy surface grain was removed and the surface leveled with a rake. Grain moistures ranged from 10.3 to 13.7 percent, grain temperatures from 72° to 94° F., and the air temperature at the time of fumigation was 80°.

A dosage of 1 gallon per 1,000 bushels did not give a complete kill of adult insects, but at both the 2- and 3-gallon dosages the kill was nearly complete (table 2). No weevils emerged from grain samples taken from the bins after fumigation.

Bromotrichloromethane-Carbon Tetrachloride Mixture

A mixture containing 10 percent of bromotrichloromethane (by volume) and 90 percent of carbon tetrachloride was tested at 3 and 4 gallons per 1,000 bushels. It was applied as a coarse spray uniformly over the surface of the grain in 3,200-bushel metal bins. The crusted

and moldy surface grain was removed before treatment. Grain moistures ranged from 11 to 12.6 percent, grain temperatures from 88° to 101° F., and the air temperature during application was 82°.

An excellent kill of adult insects was obtained at both dosages (table 2). However, samples of grain held for emergence showed that the immature stages of rice weevils were not killed.

Surface Treatments

In areas where severe infestations of the Angoumois grain moth occurred, it was observed that routine fumigations did not give satisfactory control of this insect. Even after repeated fumigations many adult moths emerged. Upon investigation it was found the surviving moths were confined largely to the surface grain.

Fumigation of the surface grain was therefore attempted with a number of materials that it was thought would be retained in large part in the first few inches. Surface treatments were made with chloropicrin in admixture with carbon tetrachloride in the proportions of 1 and 2 pounds of the chloropicrin per gallon at the rate of 1 gallon per 3,200-bushel bin. Mixtures containing 15 and 30 percent of ethylene dibromide in carbon tetrachloride were used at rates of 1 gallon per bin and 1 gallon per 1,000 bushels. As a check the 20-20 mixture of carbon tetrachloride and carbon disulfide was used at 2 and 4 gallons per bin and 2 1/2 and 5 gallons per 1,000 bushels. Two bins were usually treated with each material and at each dosage. Examination 2 weeks after treatment showed that the Angoumois grain moth was emerging from the surface grain of all bins except those treated with the ethylene dibromide-carbon tetrachloride mixtures at the rate of 1 gallon per 1,000 bushels. One live moth was seen in the bin treated with the mixture containing 15 percent of ethylene dibromide, but none in the bin treated with the 30-percent mixture.

Since a light, odorless oil applied at the rate of 2 quarts per 100 square feet was found to give a highly satisfactory kill of surface infestations of the Angoumois grain moth, it was concluded that control of such infestations by fumigation was not practical.

Spray Boom for Applying Methyl Bromide Mixtures[3/]

For the fumigation of shelled corn in quonsets with the mixtures containing methyl bromide, a method of application was developed whereby the fumigators did not have to enter the building and expose themselves to the vapors. This was accomplished by mounting a spray

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3/ This spray boom was developed with the assistance of F. D. Butcher and Max Friesen, of the Production and Marketing Administration.

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boom on a sled so that it could be pulled across the surface of the corn from outside of the building while the fumigant was sprayed in a pattern that covered the corn from one side of the building to the other (figs. 1 and 2).

Figure 1.-- Sled with spray boom mounted and connected to pump and supply tank.

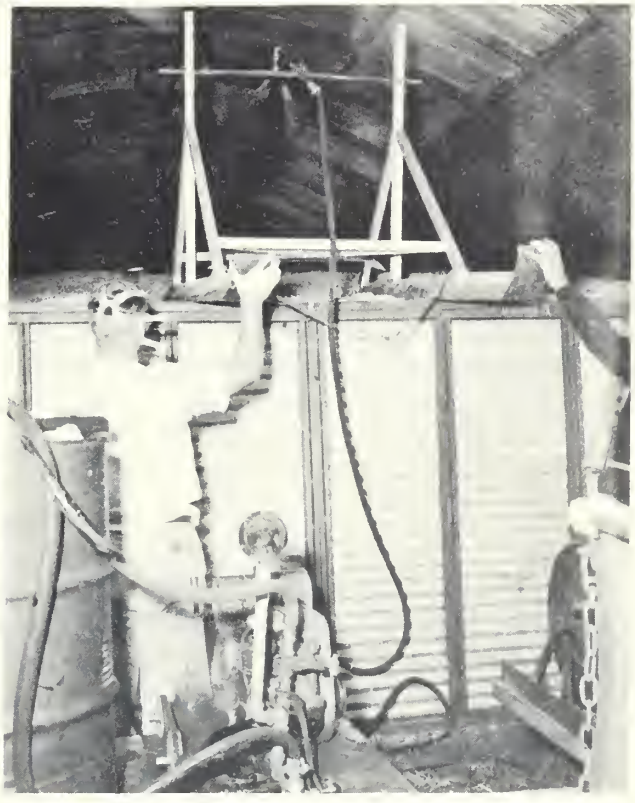

Figure 2.--Sled with spray boom on surface of corn in quonset.

The spray boom is made of a 24-inch length of 3/4-inch gas pipe bearing a nozzle with two 3/4-inch off-center nozzles. It is held firmly to a horizontal support pipe by U-bolts and strap irons to make a pipe clamp. The support pipe is of 3/4-inch gas pipe fastened by I-bolts to the uprights of the sled, 44 inches high for quonsets 32 feet wide and 49 inches high for those 40 feet wide. This pipe can be rotated by loosening the I-bolts. A hose-to-pipe fitting is attached to the front end of the nozzle pipe and a 90° street L to the back end. The nozzle is attached to the street L. The nozzle tips should be level with the support pipe and about 12 to 15 inches back of it, with the angle between the nozzles about 20° to 30°. The slotted disks in the nozzles should have the wide portion to the top or outside and the slots perpendicular or parallel to the support pipe. In this position each nozzle will cause the spray to cover the area from the center of the quonset to the opposite

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wall. The fumigant hose is connected from the other end of the nozzle pipe to the pump. This hose must be long enough to reach from the back of the quonset to the pump or the outside of the front of the building.

The wooden sled (fig. 1), weighing about 65 pounds, is constructed with runners of 1- by 6-inch boards with sections of an old steel drum on the front ends to keep them sliding. The uprights are of 2- by 2-inch boards.

A gear pump operating at 100 pounds per square inch will deliver about 2 1/2 gallons of fumigant per minute. By the use of a small winch and a wire cable attached to the sled, the boom spray can be drawn from the back to the front of the quonset at a speed that will deliver the required dosage uniformly over the grain. Such an outfit can be constructed at a nominal cost, is easily handled, and will fit into the body of a truck. A competent crew should be able to treat a quonset in about 30 minutes.

Dosages of 2 1/2 and 3 gallons of the 80-20 mixture plus 10 percent of methyl bromide per 1,000 bushels gave very satisfactory results.

Summary

An 80-20 mixture of carbon tetrachloride and carbon disulfide at a dosage of 5 gallons per 1,000 bushels has been the standard treatment for shelled corn in steel bins for several years. Unsatisfactory kills in out-of-condition corn, scarcity of materials, and the need for information regarding dosages in other types of storage structures led to further study of this mixture and other fumigants.

In metal bins the standard dosage of the 80-20 mixture was satisfactory if caked and moldy surface grain was removed before treatment unless infestations of the Angoumois grain moth were present. In wooden bins it was necessary to increase the dosage to 7 gallons per 1,000 bushels. In all bins infested with the Angoumois grain moth, it was difficult to kill this insect in the surface grain. Surface applications of fumigants proved to be impractical, but spraying with an odorless oil at the rate of 2 quarts per 100 square feet of surface grain gave a satisfactory control.

The addition of 10 percent (by volume) of methyl bromide to the 80-20 mixture of carbon tetrachloride and carbon disulfide, or to a 75-25 mixture of ethylene dichloride or propylene dichloride and carbon tetrachloride increased the toxicity to a point where satisfactory kills could be obtained with 2 gallons per 1,000 bushels in metal bins. Three gallons per 1,000 bushels of the 80-20 mixture plus methyl bromide in wooden bins and 2 1/2 to 3 gallons per 1,000 bushels in quonsets gave satisfactory results.

A spray boom mounted on a wooden sled was used to apply the methyl bromide mixture to shelled corn in quonsets from outside the building.

Other fumigants found to be highly effective for the treatment of shelled corn in metal bins were a 17-83 mixture of 1,1-dichloro-1-nitroethane and carbon tetrachloride at 2 gallons and a 10-90 mixture of bromotrichloromethane and carbon tetrachloride at 3 gallons per 1,000 bushels. The latter mixture did not kill all immature stages of the rice weevil.

Various mixtures of methyl bromide with ethylene dibromide and chloropicrin were unsatisfactory under the conditions of these tests.

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